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SPECIFICATION

1. Title of the Invention

Production Method for Oxygen Absorber

2. Claims

1. A method for producing an oxygen absorber, characterized in that the two components below are enclosed in a container having gas permeability, being brought into contact immediately prior to enclosing, or not being brought into contact before enclosing:

(A) a powder comprising iron powder and a metal halide; and

(B) a porous particulate material infused with a metal halide aqueous solution.

3. Detailed Description of the Invention

(i) Object of the Invention

(Field of Industrial Application)

The present invention relates to oxygen absorbers that are used for the storage of processed foods, farm and marine products, metal products, precision parts, fiber products and the like, and are widely used in various industries.

(Prior Art)

One quality preservation method for processed foods, farm and marine products, metal products, precision parts, fiber products and the like is a method that employs an oxygen absorber

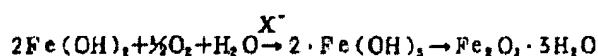
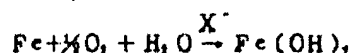
(deoxygenation agent). This method attempts to protect the target material against quality degradation due to oxygen by producing oxygen-free (or low oxygen concentration) conditions in a sealed container by absorption of oxygen with an oxygen absorber after sealing the target material, together with an oxygen absorber that is enclosed in gas permeable packaging, in a gas barrier container, specifically, a tightly sealed container such as a pouch or box produced from gas impermeable packaging material.

This method appeared on the market in Japan in 1973, and has proliferated rapidly over the past 15 years due to its simplicity and high quality retention effects. Regarding the oxygen absorber, a number of materials such as organic and inorganic materials have been proposed as the primary component for absorbing oxygen, but iron powder-based materials have become mainstream due to their superior performance and economy.

As described in the old U.K. Patent No. 553991, an iron powder-based oxygen absorber has been proposed that has active iron powder treated with hydrogen gas as the primary component, which can absorb oxygen even in a dry state.

However, iron powder that has this type of high activity carries the risk of combustion, and thus has poor practicality.

In the past, iron powder-based oxygen absorbers that have been put to actual use have all had stable iron powder as a primary component, for example, materials having water and a metal halide or the like as essential components, which employ, for example, the type of oxygen absorption reaction presented in the following chemical formulas (specifically, iron powder oxidation reaction).



(Herein, X⁻ denotes a halogen ion or the like which is a reaction accelerator.)

These types of practical iron powder-based oxygen absorbers are broadly classified into two types, self-reacting (or autoreacting) type and water-dependent type, in accordance with how the water (H₂O), which is an essential component in the reaction, is supplied to the reaction system.

Self-reacting oxygen absorbers contain water in the agent, and the oxygen absorption reaction is initiated immediately upon contact with air (strictly speaking, oxygen). This self-reacting agent is characterized by the potential for rapid oxygen absorption, even if the target to be preserved does not contain water, or has little water content.

On the other hand, water-dependent types do not themselves have water, but after being sealed in a container along with the target material to be stored (e.g., food), the very small amount of water that evaporates from the target to be stored is utilized and oxygen is absorbed. With water-dependent types, the reaction does not occur immediately as a result of simple contact with air, and they are characterized in that the workability is extremely favorable during manufacture.

The method of the present invention relates to self-reacting types according to this classification of iron powder-based oxygen absorbers.

A conventional technology related to self-reacting type iron powder-based oxygen absorbers is the method proposed in West German Patent No. 869,042 (1953) wherein a mixture of zinc, iron or other metal and activated charcoal is wetted with ammonium chloride or potassium chloride solution, and oxygen is absorbed from a gas; but the method cannot be adapted without modification as a method for the production of oxygen absorbers in small pouches that are easy to handle.

In addition, a method is proposed in the

laid-open publication of West German Patent No. 1,109,499 (1961) wherein a dry substance comprising iron powder and activated charcoal is wetted with saturated potassium chloride solution, and, after mixing well, is loaded into pouches to produce an oxygen absorber, which is sealed along with roasted coffee beans in a can; and the oxygen in the can is completely absorbed. However, with the production method for oxygen absorbers proposed therein, the oxygen absorption reaction progresses at the powder mixing stage, and there is the disadvantage that a fair amount of its performance is lost by the time it is loaded into pouches; and there is also the disadvantage that a special method such as inert gas purging must be used in order to prevent this.

A method is proposed in JP-57-314449-B as a method for solving the above problems with the production of self-reacting oxygen absorbers, wherein three essential components of the oxygen absorber, the iron powder, the water and the metal halide, are divided into the iron powder (A) and a filler (B) that is infused with metal halide aqueous solution, and are loaded in two stages and packaged together in a gas-permeable packaging material without bringing them into contact prior to packaging. With this method, the components having oxygen absorption capacity do not come into contact prior to packaging, thus there is the advantage that production is possible with substantially no attendant loss in oxygen absorption capacity; however, because the mixed condition of the essential components of the oxygen absorption reaction is poor, there is the disadvantage that the oxygen absorption rate is slow and performance varies dramatically.

(Problems to Be Solved by the Invention)

The following is a restatement of the problems with conventional methods for producing iron powder-based self-reacting type oxygen absorbers.

- (1) Extreme loss of oxygen absorption capacity occurs in methods in which the essential components of the oxygen absorption reaction are mixed, and are then loaded into a gas-permeable packaging material.
- (2) A high-quality oxygen absorber cannot be obtained with methods wherein the essential components of the oxygen absorption reaction are divided, and are loaded into a gas-permeable packaging material without allowing contact before packaging. In addition, there is significant variation in quality.

The present inventors carried out painstaking investigations towards a solution to these problems.

**(ii) Constitution of the Invention
(Means for Solving the Problems)**

The present inventors, as a result of various investigations, arrived at the present invention upon discovering that, in production methods for self-reacting oxygen absorbers, the above problems can be solved and a high-performance oxygen absorber can be reliably manufactured by utilizing a method in which a porous granular material is infused with metal halide and water together as a metal halide aqueous solution, and is enclosed in a container having gas permeability while limiting, as much as possible, contact with the powder comprising iron powder and metal halide prior to enclosing.

Specifically, the present invention relates to a method for producing an oxygen absorber, characterized in that the two components below are enclosed in a container having gas permeability, and are brought into contact immediately prior to enclosing, or without being brought into contact before enclosing:

- (A) a powder comprising iron powder and metal halide;
- (B) a porous particulate material infused with metal halide aqueous solution.

The method of the present invention is described in further detail below.

· Iron powder

The iron powder used as the (A) component is a material produced by various production methods, such as reduced iron powder, sprayed iron powder, electrolytic iron powder or ground iron powder, and these may be used individually or in combinations. In order to improve contact with the oxygen, a material is used for the iron powder that normally has an average particle diameter of 400 μm or less, preferably 200 μm or less, but if the material is too finely powdered, powder discharge will occur in the production processes such as mixing and loading, and fluidity will become poor, so a material is preferred that has an average particle diameter of 30 μm or greater.

· Metal halide

It is preferable to use, as the metal halide that is used together with the iron powder as the (A) component, a powder of one or more types of alkali metal or alkaline earth metal halides such as sodium chloride, sodium bromide, sodium iodide, potassium chloride, potassium bromide, potassium iodide, calcium chloride, magnesium chloride or barium chloride.

The amount of metal halide added is preferably 0.05 to 50 parts by weight with respect to 100 parts by weight of iron powder which is the primary

agent of the oxygen absorber, and 0.1 to 20 parts by weight is particularly preferred. If the amount of metal halide is below the above lower limit, then improvement effects will not be obtained for the oxygen absorption capacity, and above the upper limit, the metal halide component can bleed out into the gas-permeable packaging material, or other undesirable circumstances will be more likely when used in oxygen absorption reactions as an oxygen absorber that is packaged by loading into a gas permeable packaging material.

The same materials as the metal halide used for component (A) are used as the metal halide used in component (B).

The amount of metal halide that constitutes the (B) component is preferably 1 to 100 parts by weight with respect to 100 parts by weight of the porous particulate material, and 2 to 60 parts by weight is particularly preferred. If the amount of metal halide powder is below the lower limit, then the final oxygen absorption capacity of the oxygen absorber that is obtained by loading component (A) and component (B) will be poor, and there is the risk that fluidity of the (B) component will be compromised above the upper limit.

The ratio of metal halide blended in the (A) component and (B) component is 2 to 50 parts by weight with respect to the (A) component and 98 to 50 parts by weight with respect to the (B) component, and 4 to 40 parts by weight with respect to the (A) component and 96 to 60 parts by weight with respect to the (B) component are particularly preferred.

· Porous particulate material

The porous particulate material used for the (B) component is porous, and thus any material can be used, provided it is a particulate material with good water absorption capacity; but it is preferable to use a material with a BET surface area of 10 m^2/g or greater, a water absorption of 10% or greater, and a particle size of 0.1 to 5 mm.

Examples of this porous particulate material include natural zeolite, synthetic zeolite, diatomaceous earth, pearlite, activated alumina, silica gel, activated clay, magnesium silicate, sepiolite, various types of clay minerals, activated charcoal and other particulate materials.

• **Water**

The upper limit of the water used in the (B) component is preferably not greater than the saturation water absorption amount of the porous particulate material, and if it is very near the saturation water absorption amount, a condition is produced in which water is exposed at the surface of the resulting water-infused particulate material, and the fluidity of the particulate material decreases, so 0.95x or less of the saturation water absorption amount is preferred.

The lower limit of the water amount is preferably greater than 1% of the dry weight of the particulate material, and if the raw particulate material already contains greater than 1% of water, it is necessary to factor this water amount into the calculation.

• **Preparation of the (A) component**

The (A) component is prepared simply by mixing iron powder and metal halide powder, but the metal halide may be mixed with the iron powder in a condition whereby it is dissolved in water or other solvent, and then the water or solvent may be evaporated off so as to fix it to the iron powder surface.

When the metal halide is mixed with the iron powder as a powder, the material is preferably a fine powder with a particle diameter of 1 mm or less, and 200 μ m or less is particularly preferred.

• **Preparation of (B) component**

There are no particular restrictions on the method for preparing the porous particulate material that is infused with the metal halide aqueous solution, and various types of methods may be used.

For example, after introducing the porous particulate material into a mixer, a metal halide aqueous solution obtained from a prescribed amount of water and metal halide are dispersed, mixed and allowed to stand, which is repeated several times, thereby obtaining a particulate material infused uniformly with aqueous solution. Here, partitioning of aqueous solution dispersion, and repeated dispersion and mixing two or more times in this manner is also effective for obtaining a porous particulate material that is uniformly infused with metal halide aqueous solution.

It is also effective to use, as the (B) component, a material produced by adding various supplementary additives including deodorants such as activated charcoal and reaction conditioners such as calcium hydroxide and magnesium hydroxide; the supplementary additive powder is added after producing the porous particulate material with infused metal halide aqueous solution.

• **Container having gas permeability**

The container having gas permeability used in

the method of the present invention is a pouch or small molded box-shaped container that is formed from a gas-permeable packaging material; there are no special restrictions on the gas permeable packaging material; and any material may be used, provided it is a packaging material having gas permeability.

The term "gas permeable" used herein means gas permeability that can be measured as Gurley permeability described in JIS P-8117, and a preferred range for the present invention is a Gurley permeability in the range of 0.1 to 100,000 sec/100 mL.

In addition, even with packaging materials that do not themselves have measurable Gurley permeability (e.g., when the surface of the packaging is covered with a layer that is not substantially gas permeable), containers that are constituted in such a manner that passage of gas occurs from the cross section of the bonded surfaces after container molding are also containers having gas permeability that may be used in the method of the present invention.

Specific examples of the gas permeable packaging material are presented below.

1. Material formed from plastic fill [*sic*] having gas permeability and thermal adhesiveness and a gas permeable base material such as paper or nonwoven cloth

In this constitution, materials that are commonly used as packaging materials are appropriate for the paper or nonwoven cloth, and examples include, but are not limited to, papers such as Japanese paper, Kraft paper, pure white packaging paper, pure white roll paper, water-resistant paper, oil-resistant paper, oil and water-resistant paper, thin paper, and the like. In addition, any material, without restrictions, depending on the raw fiber and production method, may be used as the nonwoven cloth, provided it is a material with a melting point that is higher than the plastic film having gas permeability and thermal adhesiveness which will be describes below; and examples of raw fibers include polyester, polyamide, polypropylene, polyethylene, acrylic and the like; and materials that are produced by production methods such as dry methods, wet methods, spunbond methods and needle punch methods, or combinations thereof, can be used. In addition, materials that

are commercially available as "microporous films" produced by forming fine holes in films by various methods may also be used as the paper or nonwoven cloth in the present invention.

In addition, when the plastic film having gas permeability and thermal adhesiveness is to be produced as a package for deoxygenation agent, it constitutes the inner layer, and in general, is a material that is used as sealant; thus in order to allow heat sealing, materials are preferred that have softening points that are lower than the softening point of the base material; and materials that have air permeability due to through-holes are preferred; and materials with thicknesses of 10 to 500 μm are preferred.

Specific examples include polyethylene film, polypropylene film, ethylene vinyl acetate copolymer film, ionomer film and the like. Although there are no particular restrictions on conditions such as pore shape or number in films having fine holes as through-holes, materials are preferred in the present invention wherein the pores have diameters of 3 mm or less, preferably about 0.5 mm; the number is preferably 2 to 300 per cm^2 , with 2 to 100 being particularly preferred; the size and types of the pores can be selected in accordance with the desired deoxygenation rate.

Methods for laminating gas-permeable sealant and the gas-permeable base material that can be suitably used are wet lamination, dry lamination, hot melt lamination, thermal lamination and other common film lamination methods. In addition, when laminating a gas permeable base material and a permeable sealant, methods may also be used in which lamination is performed by providing a spacing layer in between without gluing the two layers.

2. Material also having a plastic film layer on the outside of the constitution of (1) above; specifically, a material formed from 1) a plastic film layer, 2) a gas-permeable base material such as paper or nonwoven cloth, or 3) a gas-permeable or thermal adhesive film

In this case, the plastic film of (1) is a material that has a higher softening point than the gas permeable thermal adhesive film of (3), and, for example, a film may be used that is formed from a single layer of polyethylene, polypropylene, nylon, polyethylene terephthalate, cellophane, polystyrene or polyvinyl chloride film, or two or more of these films laminated together or the like. In order to provide the plastic film of (1) with gas permeability, a material in which fine through-holes have been punched in the same manner as with the sealant layer of (3) may be used. In addition, it is also possible to use the material in a substantially gas impermeable condition without producing pores in the plastic film of (1); and, in

this case, the cross section of the heat sealing layer after formation of the package serves as the gas permeation surface.

A common film lamination method may be used as the method whereby the plastic film layer of (1) and the paper or nonwoven cloth layer of (2) are laminated, such as extrusion lamination, wet lamination, dry lamination, hot-melt lamination or thermal lamination. In addition, when laminating the plastic layer of (1) and the paper or nonwoven cloth layer of (2), a method can also be used in which lamination is carried out by providing a spacing layer in between, without gluing the two layers.

3. Material that is gas permeable and has thermal adhesiveness, such as with nonwoven cloth of polyolefin-based resin or paper produced by mixing fibers thereof

Raw material polyolefin-based resins that may be cited are polyethylene and polypropylene, and production methods include dry methods, wet methods, spunbond methods and needle punch methods; and materials can be used that are produced by a combination thereof. In addition, commercially-available materials referred to as microporous films produced by forming micropores in films by various methods may also be used as the nonwoven cloth.

Formation of the pouch for the oxygen absorber using the gas permeable packaging materials of 1 to 3 is carried out by having the thermal adhesive surfaces of the same or different materials for the gas-permeable packaging materials exemplified in 1 to 3 face inwards towards each other, and then heat-sealing the periphery.

In addition, by forming a pouch in the same manner described above using one type of gas permeable packaging material exemplified in 1 to 3 as one surface and a common gas impermeable laminated film such as polyethylene terephthalate/polyethylene or nylon/polyethylene as the other surface, it is

possible to produce a pouch for the oxygen absorber that is gas permeable on only one surface.

• Enclosing method

The (A) component and (B) component prepared in the manner described above are stored without bringing them into contact with each other, and when they are to be packaged by loading and enclosing them in the container having gas permeability, each is weighed out, loaded and enclosed in the container having gas permeability, and sealed tightly, thereby producing the oxygen absorber.

Here, there are no particular restrictions on the ratio of the (A) component and (B) component, and this is to be appropriately selected in consideration of the oxygen absorption rate, oxygen absorption capacity, production costs and the like; but normally it is preferable to make the ratio such that the water content in the (B) component is 1 to 200 parts by weight, particularly 5 to 150 parts by weight, and the total amount of metal halide is 1 to 100 parts by weight, particularly 2 to 50 parts by weight, with respect to 100 parts by weight of the total iron powder in the (A) component which is the primary agent of the oxygen absorption reaction.

When loading and enclosing in the gas permeable container, the method whereby the (A) component and (B) component are separately loaded and enclosed without bringing them into contact and are loaded so that the (A) component and (B) component are present separately in two stages in the container is a well-known method as described above; but a preferred method for providing uniformity of performance in the produced oxygen absorber is one wherein part of the tube for loading into the container from the storage tank is shared, and, in addition, a damper, stirring blade, or the like is provided, and the (A) component and (B) component that were not in previous contact are brought into contact and mixed immediately prior to loading and enclosing in the container.

[Operation]

An effect is realized whereby the following problems that self-reacting oxygen absorbers have had in the past are solved by a means whereby, in the production of an oxygen absorber from iron powder, water and metal halide, when an oxygen absorber is produced in a form whereby it can be used as-is by being enclosed in a container such as a pouch, three components, namely, the iron powder, water, and metal halide, are stored separately without being in contact simultaneously prior to enclosing in a container having gas permeability; and each is separately weighed and enclosed so as to minimize contact between the three; and metal halide and water are mixed to

produce an aqueous solution which is used for infusion into a porous particulate material, and in this condition, are mixed with the powder comprising iron powder and metal halide and used for the production of oxygen absorber.

- (1) Loss of quality and difficulties producing product with uniform quality caused by poor dispersion resulting from enclosure of the iron powder, water and metal halide without allowing contact.
- (2) Loss of performance resulting from contact of the three components, namely iron powder, water and metal halide, in the presence of oxygen.

The excellent effect described above has only now been achieved by adding metal halide to iron powder.

(Working Examples)

Hereinafter, the present invention is described in further detail based on working examples, including comparative examples.

Working Example 1

Sodium chloride powder in the amount of 0.2 kg with an average particle diameter of 150 μ was mixed with 10 kg of reduced iron powder with an average particle diameter of 80 μ for 15 min with a V-shaped mixer, and the resulting powder was used as the (A) component.

Separately, particulate sepiolite (magnesium silicate mineral, fired at 200°C) having a particle diameter of 0.5 to 3 mm was introduced into a V-shaped mixer, sodium chloride solution produced by dissolving 1.2 kg of sodium chloride in 4.6 kg of water was introduced therein, and after mixing for 15 minutes, letting stand for 8 hours, mixing for 15 minutes again, letting stand for 16 hours, mixing for 15 minutes again, letting stand for 8 hours (32 hours elapsed from the start), and mixing for 15 minutes, a particulate material with good fluidity was obtained. The resulting powder was used as the (B) component.

The (A) component and (B) component were each weighed in the amount of 1.5 g, and were loaded and sealed in two stages by introducing each separately into a pouch with a size of 40 x 50 mm produced from gas-permeable packaging paper formed by laminating paper and porous polyethylene (Gurley permeability about 2000 sec/100 mL). Five of the same items were produced.

The resulting oxygen absorber was sealed in a gas barrier bag along with 500 cc of air, and allowed to stand at 20°C, whereupon the change in oxygen concentration in the system was successively analyzed, and was found to be 4.1%, 3.5%, 3.8%, 3.2%

and 2.9% (average 3.5%, standard deviation $\sigma_{n-1} = 0.47$) after 8 h, and 0% for all of them after 16 h.

Comparative Example 1

A particulate material was used as the (B) component, which was obtained in the same manner as in Working Example 1, with the exception that the added amount of sodium chloride powder in the production method for the (B) component in Working Example 1 was 1.4 kg.

Only iron powder used in Working Example 1 was used as the (A) component.

This (A) component and (B) component were used, and with this exception, five oxygen absorbers were produced in exactly the same manner as in Working Example 1. The amounts of the components in the resulting oxygen absorbers were about the same as in Working Example 1, and only the sodium chloride distribution condition was different (in Working Example 1, the sodium chloride was introduced into both the (A) component and (B) component, but in this example, sodium chloride was introduced only into the (B) component).

The oxygen absorption capacity of the resulting oxygen absorber was measured by the same method as in Working Example 1, and was 7.0%, 4.5%, 5.1%, 7.0%, and 3.8% after 8 hours (average 5.5%, standard deviation $\sigma_{n-1} = 1.46$), and 0% for all of them after 16 hours.

The average value for the residual oxygen concentration after 8 hours was higher than the average value in Working Example 1, and the data dispersion (standard deviation) was also high.

Working Example 2

Sodium chloride powder in the amount of 0.2 kg, with an average particle diameter of 150 μ was mixed with 10 kg of reduced iron powder with an average particle diameter of 80 μ for 15 minutes with a V-shaped mixer, and the resulting powder was used as (A) component.

Separately, 10 kg of particulate diatomaceous earth with a particle diameter of 0.15 to 1 mm (product obtained by fusing mixture firing at 1200°C) was introduced into a V-type mixer, sodium chloride aqueous solution produced by dissolving 2.1 kg of sodium chloride in 8.0 kg of water was then introduced therein, and mixing was performed in the same manner as in Working Example 1 to obtain a particulate material with good fluidity. The resulting powder was used as the (B) component.

1.5 g each of the (A) component and (B) component were weighed, and were loaded and sealed in two stages by introducing each separately into a pouch with a size of 40 x 50 mm produced from gas-permeable packaging paper formed by laminating paper and porous polyethylene (Gurley permeability about 2000

sec/100 mL). Five of the same items were produced.

The resulting oxygen absorber was sealed in a gas barrier bag along with 500 cc of air, and allowed to stand at 20°C, whereupon the change in oxygen concentration in the system was successively analyzed, and was found to be 4.0%, 4.2%, 4.7%, 4.6% and 5.0% after 6 hours (average 4.5%, standard deviation $\sigma_{n-1} = 0.40$), and 0% for all of them after 16 hours.

Comparative Example 2

A particulate material (diatomaceous earth) was used as the (B) component, which was obtained in the same manner as in Working Example 2, with the exception that the added amount of sodium chloride powder in the production method for the (B) component in Working Example 2 was 2.3 kg.

Only iron powder used in Working example 2 was used as the (A) component. This (A) component and (B) component were used, and with this exception, five oxygen absorbers were produced in exactly the same manner as in Working Example 2. The amounts of the components in the resulting oxygen absorbers were about the same as in Working Example 2, and only the sodium chloride distribution condition was different (in Working Example 2, the sodium chloride was introduced into both the (A) component and (B) component, but in this example, sodium chloride was introduced only into the (B) component).

The oxygen absorption capacity of the resulting oxygen absorber was measured by the same method as in Working Example 2, and was 4.5%, 6.2%, 5.7%, 6.5%, and 7.0% after 6 hours (average 6.4%, standard deviation $\sigma_{n-1} = 1.39$), and 0% for all of them after 16 hours.

The average value for the residual oxygen concentration after 6 hours was higher than the average value in Working Example 2, and the data dispersion (standard deviation) was also high.

(iii) Effect of the Invention

The present invention allows constant production of a product with superior quality without a decrease in quality of the self-reacting oxygen absorber; and also contributes greatly to the deoxygenation agent production industry and various industries that employ deoxygenation agents.

Applicant name:

Toa Gosei Chemical Industries